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Report

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NOVEMBER 1952 SYMPOSIUM ON INFRARED SPECTROSCOPIC
TECHNIQUES HELD IN THE INSTITUTE OF PHYSICAL CHEMISTRY,
UNIVERSITY OF FREIBURG IM BREISGAU (FRENCH ZONE)

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U. Schied, W. Nothdurft, V. Kessler

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or prepared for publication.)

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17-18 NOVEMBER SYMPOSIUM (11 REPORTS) ON INFRARED
SPECTROSCOPIC TECHNIQUES HELD IN THE INSTITUTE OF
PHYSICAL CHEMISTRY, UNIVERSITY OF FREIBURG IM
BREISGAU (FRENCH ZONE)

Comment: The following represents information on west German reports on infrared techniques; the information also includes title of report, author's name, number of pages of the original reports. Preceding these reports, numbered 1 to 11, is the general program, designated by 0 outlining the order of events; as far as possible the reports are given here in the order apparently listed in the program -- thus Mutter's report is given first here since his name is mentioned first in the list of speakers of the first day of the symposium, etc.

0. "SYMPOSIUM ON INFRARED SPECTROSCOPIC TECHNIQUES IN THE INSTITUTE OF PHYSICAL CHEMISTRY, UNIVERSITY OF FREIBURG IM BREISGAU, HEBELSTRASSE 38, HELD FROM 17 TO 18 NOVEMBER 1952"

Comment: 1 page in original

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Monday, 17 Nov 1952	I. The Spectrum and it's Interpretation.
0900 - 1230	Evaluation of frequency and intensity. Integration of absorption bands. Qualitative and quantitative analysis. Reproduction of spectra. Catalog and atlas of spectra. Bibliography of literature.
Scheduled reports by Mutter, Oswald, and Schmid.	
Dinner together at Zahringer Grill, Bertholdstrasse.	
1500 - 1800	II. Techniques of Photography.
	Preparation of samples of substances in gaseous, liquid or solid states. Gas vessels; heated and cooled vessels. Dissolving agents. Infrared microscopy. Measurement of coating thickness.
Scheduled reports by Schiedt, Nothdurft, Funck.	

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Tuesday, 18 Nov 1952

0845 - 1230

III. The Spectrometer.

Modern and future development. Calibration.

Adjustment. Efficiency and ability for reproduction.

Scheduled reports by Hausdorff, and Schmid.

Dinner

During the symposium practical demonstrations and visiting of the institute.

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1. DETERMINATION OF HEXACHLOROCYCLOHEXANE IN TECHNICAL CRUDE PRODUCTS.
EXAMPLE OF A QUANTITATIVE ANALYSIS

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Rudolf Mutter (Freiburg im Breisgau).

Comment: 1½ page in original; completely translated here.

Considering the great accuracy which may be attained in the specific determining method of infrared analysis, we had to investigate experimentally what accuracy is practicable, particularly in the determination of gamma-isomers of technical crude products. Because of the occurrence of epsilon-isomer only in very small concentration with low solubility of beta-hexachlorocyclohexane in carbon disulfide, only the system of three substances alpha, gamma, delta - $C_6H_6Cl_6$ was investigated. In order that no gamma-hexachlorocyclohexane should be lost during preliminary treatment of the samples, the samples were dissolved directly in carbon disulfide and their spectra taken in the Perkin-Elmer infrared spectrometer Model 21 with optical system of rock salt ($100 \text{ mg}/5 \text{ cm}^3$, $d = 1 \text{ mm}$). It is not advantageous to use stronger concentrations, because we reach the limit of solvability, nor to use thicker coating, because of the resulting stronger absorption by the dissolving agent within the range of measurement ($7 - 15.5\mu$).

The crude products obtain disturbing impurities of unknown composition (chlorobenzols and higher chlorinated cyclohexanes etc.) which add their absorption bands to the spectra of the main substances. In order to recognize and to eliminate individual disturbing bands, we use all key bands in the analysis.

gamma:	7.85	$\times 10.34$	$\times 11.00$	$\times 11.84$	$\times 14.54$	$\times 14.98$
alpha:	7.64	7.95				
delta:	7.55	8.13	10.17	13.23		

These are used to compute the components in the first approximation using three-term determinants [cf. W. Siebert, Z. Elektrochem. 54 (1950)]. Using the computed parts of isomers, we determine the background produced by the superposition of bands for the key bands and we subtract it from the measured

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curve $\log i_0/i$. If we chose for the gamma-content the 5-key bands above (x) we obtain in general discrepancies of only ± 1 to 2%; and in very unfavorable cases, up to 3% abs. In the case of pure products (enriched) the values obtained from the 5 key bands deviate from ± 0.5 to 1% abs.

Noted sources of errors were specially indicated:

- 1) Gases (dissolving agent) which diffused into the apparatus because of intrinsic absorption may disturb the band to be measured and even repress it entirely.
- 2) Certain samples show, starting with about 11μ , an increasing continuum which is not due either to H_2O or HCl, as proved by investigation. It is deceiving in the case of bands of longer wave lengths, in indicating an allegedly higher content of gamma-isomer, and therefore should be compensated. An estimate is possible in that the apparent increase of content of gamma-isomer is smaller in bands of higher extinction than with smaller epsilon.

For accurate data the behavior of the curve should be compared with spectra of weighed mixtures of pure substances, which allows one to recognize the effect of disturbing bands, as of new bands or shifts. It follows from the method of the determination that the relative accuracy is increasing with higher content of gamma - hexachlorocyclohexane.

2. DETERMINATION OF INTENSITY OF INFRARED ABSORPTION BANDS

Friedrich Oswald (Freiburg im Breisgau)

Comment: Abstract translation; 1½ page in original. The part omitted here was the description of American methods.

In the case of evaluation of infrared absorption spectrograms for quantitative analysis the total absorption $A = \int \log i_0/i d\nu = c \cdot d \int \epsilon d\nu$ is a better measure of the intensity of an absorption band than its maximal height $\log (i_0/i)_{max}$, because the total absorption depends only slightly on the operating conditions (spectral slit width, scattered light). Various procedures for determination of A are summarized and discussed:

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- 1) Bourgin, Phys. Rev. 29, 794 (1927).
- 2) D. A. Ramsay, J. Am. Chem. Soc. 74 (1952).
- 3) Logarithmic scale
 - a) Logarithmic scale paper facilitates reading of points and plotting of the ordinate values i/i_0 as extinction values of $\log i_0/i$; but only in the case where the line for $i = 0$ and $i = i_0$ correspond to the lines infinity ∞ and zero 0 of the coordinate system.
 - b) In other cases the coordinates of the curve and of the i_0 - line have to be measured in logarithmic scale and deducted starting from the 0-line.
 - c) An aligning table with 50 logarithmic scales of various lengths units substitutes for subtraction.
 - d) A drawing net with two colored superposed divisions (one linear and the other logarithmic) enable direct redrawing by points of the recorded curve into a $\log i_0/i$ - curve, without writing down of intermediate values.
- 4) The auxiliary means described in b to d are used in this institute.
- 5) H. Janeschitz-Kriegl, Graz, Chem. Ing. Techn. 24 (1952). Drawing equipment with logarithmic spiral enables a continuous redrawing of transparency curves into extinction curves.
- 6) E. A. Francis, J. Chem. Phys. 18 (1950). A logarithmic integrator supplies simultaneously the band content A.
- 7) The IPM- OTt integrating equipment [cf. Walther und Dreyer, Naturwiss 36 (1949)] performs the mentioned operations by remote control, after having scanned the absorption curve.

For practical means a compromise between accuracy and expenditure of time and equipment should be found; thus the methods 3 to 5 seem to be the best for application.

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3. THE DOCUMENTATION OF INFRARED ABSORPTION DATA

R. Mecke and E. D. Schmid (Institute of Physical Chemistry, University of Freiburg im Breisgau).

Comment: Original was 12 pages; which are abbreviated here.

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For the past several years self-recording infrared spectrographs have been finding more and more extensive use in research and techniques. These developments produced a considerable crisis in documentation of infrared absorption spectra, because of the ever increasing number of spectrograms. The spectrograms are disseminated over numerous institutes and research centers and their number is difficult to evaluate; it may well exceed 10,000. Therefore the creation of an institute that is able to collect and classify available and incoming materials and to supply it to interested people becomes of urgent need.

In the present work an attempt is made to outline the viewpoints which should be taken into consideration in the development of a method for collection of data, and the ways to the solution of the problem of documentation. A consideration of methods already devised may clarify their application. Before describing the difficulties and requirements in infrared spectroscopy, one should first give a general sketch of the situation prevailing today in documentation. It will facilitate to chose from this sketch all viewpoints which could give a satisfactory solution of the problem of documentation of infrared spectroscopy.

The sources of international information are:

1. Periodicals. 2. Handbooks. 3. Current documentation, as e. g. represented by "Chemisches Zentralblatt", "Chemical Abstracts" etc, which are excellent informative sources; due to their wide scope, however, they are unable to give exhaustive answers.

The punched card is an auxiliary means of documentation of infrared absorption data. Comment: The original document is describes in detail American punch-card systems and IBM cards; also the "compound card" and the "bibliography card". Refers as a source of information: Committee on Infrared Absorption Spectra, E. Carroll Gratz, Secretary National Bureau of Standards,

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4. THE DOCUMENTATION OF INFRARED ABSORPTION SPECTRA.

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E. D. Schmid (Freiburg im Breisgau)

Comment: The original is 1 page, which describes documentation centers in USA.

Europe does not yet possess a central institution for gathering and distribution of infrared absorption spectra, like that of the National Bureau of Standards in USA, nor is the creation of such an institution planned in the near future.

5. THE EFFICIENCY OF THE INFRARED SPECTROPHOTOMETER MODEL 21 OF THE AMERICAN PERKIN-ELMER CORPORATION.

E. D. Schmid (Freiburg im Breisgau)

Comment: The report refers only to American information on the subject. The original document was 1 page.

6. PROCEDURE FOR INVESTIGATION OF SOLIDS BY EMBEDDING INTO POTASSIUM BROMIDE

U. Schiedt (Tubingen)

Comment: Original report was 1 page, which is given entirely here.

The difficulty of investigation of solid or hardly soluble substances is well known. All procedures to deposit substances in the solid state on a suitable carrier involve usually strongly scattering layers, without even mentioning the difficulty of determining the concentration and thickness of the coating. Recent results of embedding in nujol, perfluoro-kerosene etc were not satisfactory because of the intrinsic absorption of the embedding materials and the generally impossible determination of concentration; however, there has been devised a new already-published procedure cf. U. Schiedt and H. Reinwein, Z. Naturforschung 76 (1952) of embedding into potassium bromide, which becomes plastic under high pressures. A quantitatively prepared mixture containing potassium bromide and the substance to be examined is pressed in a precision press on to a glass clear plate. In order to avoid presence of air, the equipment should be evacuated during the compression. The pressure is at least 5 tons/cm² on the plate surface. A high as possible

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dispersive distribution of the substance in the compressed mixture is necessary in order to obtain reliable spectra which allow a quantitative evaluation. This is achieved by strong grinding of the mixture in a grinding machine. The efficiency of the grinding may still be increased by a previously applied process similar to drying by freezing.

7. FLUID VESSELS

W. Northdurft (Hochst)

Comment: Complete translation, although brief, is given below.

Fluid vessels made from circular hard-salt plates were described. Two capillaries made of nickel, V2A-steel or glass were adjusted into slightly conical borings in one of the two hard salt plates. The vessels are impervious without sealing.

Recorded curves were demonstrated, they were written by ball pens instead of ink pens.

8. APPLICATION AND CONSTRUCTION OF HEATING AND COOLING CELLS

Ernst Funck (Freiburg)

Comment: Complete translation of original 1 page report is given below.

The purpose of heating and cooling cells is to investigate substances in states different from those at normal temperature and to find out the resulting changes in the spectra. Moreover they are necessary for studying equilibrium, reactions of various type (association, isomerism, and real chemical reaction), and changes in the rotational structure of gaseous bands with temperature and for identifying difference bands.

A survey of the available literature [a good bibliography may be found in: Lord, McDonald and Miller, J.Opt. Soc. Am. 42 (1952)] shows the predominance of some less typical constructions. Gaseous heating cells are usually constructed from glass cylinders with sealed windows, which are set into a closed electric oven with a corresponding window. In this way the whole cell including

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the window is evenly heated. In the case of cells for liquids, the cell's frame is extended into a tube on which a heating coil is wound directly. In the case of cooling cells the usual cooling media are applied, such as dry ice, liquid air, etc. Less seldom used are cooling thermostats with circulation or refrigerating machines. The proper cells have to be set into an evacuated vessel with windows in order to prevent deposition of water on the cell or on the solid sample and to support the heat transfer. Cells for fluids and for solid bodies are set on the bottom of the vessel, which are filled with a cooling agent and put in from above into the evacuated vessel. Gas cells are set into the container in a way that the walls around are in contact with the cooling agent. The container is entirely surrounded by vacuum. In the case of cooling with liquid helium a screen also is provided between the external wall and the container with liquid helium; and the screen is cooled with liquid nitrogen. A more obsolete construction of cooling cells is the reflection cell, in which a ray is reflected by the polished metallic plate which holds the substance.

A heating vessel for gases, which was built in the institutes, consists of a 9-cm long gas cylinder on which an electric heating coil on asbestos cloth is directly wound. The windows of rock salt on both ends are heated through heating wires 0.1 mm thick, which are fixed at an 3 mm interval from the external side of the windows. By these means the vessel is evenly heated and the ~~deposit~~ on windows of fluids evaporated in the vessel is avoided. For the temperature reading a thermometer or a thermoelement is set into a special cut. The cell may be used without change of the path of radiation in the Perkin-Elmer Model 21.

9. NEW DEVELOPMENTS IN THE PERKIN ELMER CORPORATION

H. Hausdorf (Norwalk, Connecticut, USA)

Comment: Original report was 2½ pages. No translation is given here.

10. PRODUCTION OF EXTREMELY THIN LIQUID LAYERS FOR MEASUREMENTS OF ABSORPTION IN ULTRAVIOLET.

V. V. Keussler (Freiburg)

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Comment: Complete translation is given below.]

A thin ring-shaped metallic coat (aluminum) is deposited by evaporation in high vacuum on quartz plates, which is cut with high accuracy, forming the absorption vessel and serving as interval ring. In this way layers of a few micron thickness down to 0.94 micron were obtained. Measurements of light intensity transmitted through an empty vessel by means of an electric light Beckmann spectral photometer allow determination of great accuracy (over 1%) of the plate interval by means of the interval of interference strips in the spectrum. It is possible to produce still thinner coatings than those obtained and to measure them by the same method. By application of the thinnest coatings obtained by this method it became possible to make the first measurement of the ultraviolet absorption spectrum of benzol.

11. REPORT ON NEW INFRARED SPECTROSCOPY IN USA

Jean Lecomte (Paris)

Comment: The original report was 1 page. It is not translated.]

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